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# A SURVEY OF SOLID LUBRICANT TECHNOLOGY



## TECHNICAL REPORT

Peter Martin, Jr.

March 1972



RESEARCH DIRECTORATE ·

WEAPONS LABORATORY AT ROCK ISLAND

RESEARCH, DEVELOPMENT AND ENGINEERING DIRECTORATE

U. S. ARMY WEAPONS COMMAND

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# RESEARCH DIRECTORATE WEAPONS LABORATORY AT ROCK ISLAND RESEARCH, DEVELOPMENT AND ENGINEERING DIRECTORATE

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A SURVEY OF SOLID LUBRICANT TECHNOLOGY

Peter Martin, Jr.

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### **ABSTRACT**

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New developments in the areas of solid lubricant powders, bonded films, and self-lubricating composites have been reviewed along with new application rechniques by the Research Directorate, Weapons Laboratory at Rock Island.

Recently synthesized lubricant solids include sulfides, selenides and tellurides of heavy metals. Some of these new materials have been incorporated into oils, greases, dry films, and composites with resulting improved properties such as lower friction coefficients, greater oxidation stability, and chemical stability.

The high temperature and wear life properties of bonded solid film lubricants have been improved by use of new organic binder materials such as polyimides, polybenzimidazoles, and polybenzothiazoles. The use of inorganic binders of silicates, phosphates, and fluorides has extended the temperatures to 1200°F with some impairment of performance at room temperatures.

New techniques for appling solid lubricants are those of plasma spraying, electrophoresis, electrodeposition, vacuum deposition, and ion plating.

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Self-lubricating composites, recently developed, are of the metal- and plastic-based types. Metal-based components have higher strength and higher temperature capability as compared with that of the plastic composites.

Plastic composites have been improved for higher strength and heat stability by use of aromatic polyimides. Poly-tetrafluoroethylene-based composites have been improved in high strength properties by use of glass and carbon fibers.

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### INTRODUCTION

A literature survey dealing with solid lubrication technology has been made by the Research Directorate, Weapons Laboratory at Rock Island. This technology is approximately 20 years old. Impetus for the growth of this technology has come from the lubrication requirements of jet aircraft. During the last 10 years, lubrication requirements for space applications have provided additional incentive for the advancement of solid lubrication. The general effort expended to advance solid lubrication is small when compared to that expended on liquid lubrication; however, progress made on solid lubricances during the last 20 years has been substantial.

Solid lubricants are usually considered for use only where oil and grease lubricants cannot be used. These areas include high loads, extreme temperatures, high vacuum, and adverse environments. Solid lubricants, however, can also be used under moderate conditions in areas commonly lubricated with oils or greases. These lubricants can be in the form of powders and bonded films, or additives in oils and greases, and in the form of an integral part or composite.

The use of solid lubricants provides the following advantages: (1) dry airborne dirt does not collect, (2) stability at high temperatures, (3) high load capacities, and (4) application by which simpler design is permitted by elimination of the need for oil seals and pumps.

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The use of solid lubricants entails, however, the following disadvantages: (1) the friction coefficient is generally higher than that of the fluid lubrication, (2) bonded film coating has a finite wear 1 fe, and (3) this coating is not applicable to high-speed areas, such as automotive engines.

However, the outstanding characteristic of solid lubricants is the concept of permanent lubrication for the life of the system. This feature has definite potential use in the area of weapons in which periodic maintenance of lubricating and cleaning are required. The application of solid lubricants to weapons would result in reduced maintenance, reduced wear, dust-free lubrication, and elimination of the "wear in" period.

However, selection of the proper solid lubricant system for a specific application is a complex lubrication engineering task. This effort has been made more difficult because of the rapid development of solid lubrication technology. Therefore, a systematic survey is needed to indicate the current state of the art and to provide a basis for the optimus selection of solid lubricants for future weapon applications.

### APPROACH

ture sizely and comprises three parts. Part I deals with sol appricant powders, Part II involves bonded film solid lubricants, and Part III concerns information on solid lubricating composites.

### ISCUSSION

Part I. Solid Lubricant Powders

Three solid lubricant powders - graphite, molybdenum disulfide  $(MoS_2)$ , and polytetrafluoroethylene (PTFE) - have been given the most attention and application.

Graphite lubricates best in the presence of moisture. This limits its applications since the presence of water can cause galvanic corrosion when other metals are in contact. Molybdenum disulfide oxidizes readily at temperatures above 600°F; this limits its use at high temperatures. Polytetrafluoroethylene produces a low friction coating but cold flows under high loads, and decomposes above 600°F.

The limitations of these materials has stimulated research toward the synthesis of other solid lubricants. The research was aimed at the production of materials with lamellar, hexagonal structures similar to molybdenum disulfide. These new materials were sulfides, selenides, and tellurides of heavy metals. These materials were produced by Boes<sup>1</sup>, Magie<sup>2</sup>, and Devine<sup>3</sup>. The new materials were characterized by having greater exidation stability, electrical conductivity, and chemical stability than the standard solid lubricants.

The air stability and coefficient of friction values for the new materials and  ${\rm MoS}_2$  are given in Table I.

TABLE I
NEW SOLID LUBRICANTS

Compound	Air Stability (°F)	Coeff. of Friction
Molybdenum disulfide (MoS <sub>2</sub> )	660	.18
Molybdenum diselenide (MoSe <sub>2</sub> )	750	.17
Tungsten disulfide (WS <sub>2</sub> )	820	.17
Tungsten diselenide (WSe <sub>2</sub> )	660	.09
Niobium disulfide (NbS <sub>2</sub> )	790	.08
Niobium diselenide (NbSe <sub>2</sub> )	660	.12
Tantalum disulfide (TaS <sub>2</sub> )	1110	.05
Tanta!um diselenide (TaSe <sub>2</sub> )	1070	.08
Arsenic thioarsenate (AsAsS <sub>4</sub> )	750	
Arsenic thioantimonate (AsSbS <sub>4</sub> )	750	~ ~ ~
Graphite fluoride (CF.,)	750	. 05

For actual applications, these powders may be used in other forms. They can be (a) dusted or burnished and impregnated into the mating surfaces, (b) suspended in a controlled stream of carrier gas, (c) dispersed in a binder to form a thin film, (d) dispersed in a metal or plastic matrix (composite), and (e) formulated into oils and greases as extreme pressure additives.

Magie  $^2$  has reported on the work performed with Ag-NbSe $_2$  compacts. The compacts were evaluated for use as commutator brushes. These brushes gave considerably better electrical properties, yet gave wear rates comparable to Ag-MoS $_2$  brushes.

WSe<sub>2</sub> has been combined in powdered form with galluim-indium alloy by Boes<sup>2</sup>. This work resulted in a compact for use as self-lubricating members in high speed - high temperature ball bearing systems. This material is of considerable interest since it resists oxidation at temperatures much higher than pure WSe<sub>2</sub>.

Hubbell<sup>5</sup>, developed compacts with cobalt containing WS<sub>2</sub>. These materials performed well as separator materials for use in the lubrication of ball bearings. The materials developed have potential for widespread use in many different machine element applications such as sleeve and spherical bearings, and gears and tracks.

Two sulfides, AsSbS, and AsAsS,, were prepared and examined as chemical additions for diester lubricating grease and organic resin solid film lubricants by Devine The arsenic sulfides exhibited wear resistance superior to  $\text{MoS}_2$  in similar concentrations in the base grease. However, the arsenic sulfides showed no improvement when compared with  $\text{MoS}_2$  in resin-bonded solid film.

Fusaro studied the friction and wear life of burnished films of graphite fluoride. Test comparisons indicated that the wear life of  $CF_X$  films exceeded that of graphite or MoS<sub>2</sub>. Moisture seems to benefit the wear life of graphite fluoride, but it is not essential for lubrication.

Additional studies on graphite fluoride have been conducted by Petronio?. The solid lubricant was formulated with silicate and epoxy-phenolic binders, and with diester-based grease. Preliminary studies indicated that graphite fluoride extended the wear life of bonded films as compared with graphite-bonded films.

### Part II. Bonded Solid Lubricants

### a. Resin and Ceramic Bonded Films

Bonded or "dry film lubricant" is used to designate a series of lubricating materials consisting of a thin film of solid lubricant (MoS<sub>2</sub>, graphite) attached to the surface to be lubricated with an adhesive material. This adhesive material may be organic (phenolic, epoxy, polyimide resins) or inorganic (sodium silicate, aluminum phosphate) in nature. The organically bonded dry film lubricants can be subdivided into heat-cured and air-cured, dry film lubricants, and the inorganic films can be subdivided into nonceramic and ceramic binding. Dry film lubricants may be applied by the most common methods of dipping, spraying, and brushing. New methods of application comprise electrophoretic deposition, plasma-spraying, and vacuum-sputtering. These new methods appear feasible, but are still in the developmental stage.

A list of recently developed dry film lubricants, with the method of application, is included in Table II. These new materials are a result of the emphasis on hightemperature applications and improvement of endurance life THE THE THE STANDING STREET OF THE STANDING STANDING STANDING STREET STANDING STANDI

In the past, organically bonded solid lubricants have been temperature-limited by the binder materials. However, new resins for use as binder materials have now been developed that are more thermally stable than  $MoS_2$ . The use of these new binders has been examined by several investigators and is snown in the first four films of Table II.

A high-temperature resin polyimide (PI) was used as the binder or new solid film lubricant designated as MLR-2. The work, reported by Campbell<sup>8</sup>, showed that polyimide-bonded lubricant films exhibited usable wear life to 700°F.

McConnell has worked with polybenzo+hiazole (PBT) and has indicated that films (AFSL-26) bonded with the PBT resin have superior wear performance above 500°F.

Polybenzimidazole (PBI) is another high-temperature resin developed by Hopkins for use as a bonding agent. The new solid film lubricant AFSL-32 has a 2n0 additive that is beneficial to wear life. This beneficial effect by the addition of an oxide has been explained by an oxide interaction hypothesis. In this

TABLE II

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# BONDED SOLID FILM LUBRICANTS

# Sprayed, then heat-cured 2 hrs. at 180°F, followed by 2-15 hr. at 400°F. Sprayed, then heat-cured 1 hr. at 150°F, 2 hr.at 200°F, 8 hr. at 400°F. Sprayed, then heat-cured 1 hr. at 200°F, followed by 1 hr. at 575°F. Sprayed, then heat-cured 1 hr. at 200°F, followed by 1 hr. at 600°F. Sprayed, then heat-cured 1 hr. at 200°F, followed by 1 hr. at 575°F. Sprayed, then heat-cured 1 hr. at 95°C, 200°C, 300°C, 1 hr. at 806-1100°C. Sprayed, then cured at room temp. for 24 hrs. Sprayed, then heat-cured 1 hr at 95°C, 200°C, 300°C, 1 hr. at 800-1100°C. Application Method $MoSe_2 + TaS_2 + graphite$ $MoS_2 + Sb_2O_3 + prefused$ $MoS_2$ + graphite + Au $MoS_2 + Sb_2O_3 + ZnO$ $MoS_2 + Sb_20_3$ Additives $MoS_2 + Sb_20_3$ $MoS_2 + Sb_20_3$ BaF2:CaF2 Materials methylphenylsi;icone aluminum phosphate aluminum phosphate polybenzothiazole polybenzimidazole polybenzimidazole sodium silicate Binder

polyimide

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concept, the friction and the wear of bonded films, simple transfer films, and composites of  $MoS_2$  are decreased by use of low melting oxides. These exides are combined easily to form desirable eutectics with molybdenum oxides. The results of Hopkins' study and those of other investigations seem to support the hypothesis for oxide interaction.

All new resins cited above required a heat-curing process; however, a methyl phenyl silicone resin was selected by Benzing 12 as a binder for an air-cured solid film lubricant. The purpose of this work was to obtain a solid film lubricant that could be applied in the field and that would retain performance characteristics comparable to those of heat-cured films. The material was designated as AFSL-41, and could be used at temperatures to 700°F. This material also has the unique capacity to perform well on titanium surfaces in addition to the more conventional steel alloys.

The high temperature inorganic solid lubricants are an extension to the resin bonded films. These materials are intended for use at temperatures from 500°F to 1200°F and above. Salt-based or ceramic binders are used to give greater temperature resistance than that of resins, and usually contain solid lubricants which are more thermally stable than graphite or MoS<sub>2</sub>. These solid lubricants can be lead oxide, lead sulfide, calcium fluoride, gold, silver, tellurides, and selenides.

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The nonceramic binders are usually water soluble silicates and phosphates which produce a hard coating that tends to be brittle when cured. In general, these nonceramic binders can be used at temperatures from 300 to 1000°F. Hopkins¹² reports on an inorganic nonceramic solid film lubricant developed for service in space environments. This material contains MoS¹, graphite, and gold dispersed as the solid lubricants in a sodium silicate binder. The material had good high load-carrying capacity, and the films exhibited lower friction coefficients in vacuum than in air.

A continuation of this development was the work by Wieser<sup>12</sup> on an improved solid film consisting of MoSe<sub>2</sub>, TaS<sub>2</sub>, and graphite in an aluminum phosphate binder. Experiments were performed to find out whether the wear life of the original film can be improved by an overcoat applied by electrophoretic deposition. The second coat that was deposited consisted of MoS<sub>2</sub> and B<sub>2</sub>O<sub>2</sub>. Results have indicated an improvement in the wear life at 1000°F.

The inorganic ceramic bonding agents are glasses that melt when heated. When cooled, these glasses serve as a bonding agent for the dispersed solid lubricant. Although ceramic-bonded materials have higher coefficient of friction and shorter wear life than resin-bonded materials at room temperature, these materials generally exceed the performance of a resin film at higher temperatures (>1000°F). However, subjecting ceramic bonded film to high speeds at room temperature will result in high temperatures generated at the contact zone; this could result in a performance comparable to resin-bonded films. One problem in the use of ceramic-bonded materials is that of the thermal expansion of the cured coating. This must be matched closely with the expansion of the base material to prevent fracturing or flaking.

Extensive work by Sliney<sup>1</sup> resulted in the establishment of friction and wear characteristics of fluorides as ceramic binders. The films made with these binders exhibited the following disadvantages: (1) two operations are required for preparation of the film, (2) high-fusing temperature is detrimental to many alloys, and (3) films had to be applied thick, then ground to achieve desired film thickness.

The study of fluoride-based films was extended by Lavik<sup>15</sup>. The performance of these films (AFSL-28) indicated that operation in the 750°F to 1450°F range may be expected to give relatively long wear life at comparatively high loads. For operation between room temperature and 750°F, long-term, light-load conditions can be best achieved with the addition of 10 per cent gold powder to the films.

In a more recent study by Hopkins,  $^{16}$  prefused fluorides were combined with MoS<sub>2</sub>, Sb<sub>2</sub>O<sub>3</sub>, and polybenzimidazole. The addition of fluorides enhanced the wear life performance of the original PBI-MoS<sub>2</sub> films (AFSL-27) in the range from room temperature to 600°F.

### b. New Bonding Methods

Solid film lubricants that must be cured for a certain time at elevated temperatures, particularly fused ceramic bonded films, are not feasible for production line or field application because of the time and the facilities required for curing. In addition the high curing temperature may affect the mechanical properties of the substrate metal or alloy. Therefore, the most desirable processes are those in which films can be applied and cured at the same time or processes in which deposition of the films

results in greater energy of adhesion.

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The process with the use of a plasma spray qun for the application of various solid lubricant films was described by Hopkins. 16 This process involved the successful application of metal, resin, and ceramic solid lubricants. However, the partial thermal degradation of MoS, to elemental molybdenum and MoO<sub>3</sub> during plasma-spraying led to further studies by Kremith<sup>17</sup> to reduce thermal deg-Kremith's approach involved the use of metal composite powders which not only provided thermal protection for the solid lubricant but also decreased material segragation during the spray operation. The composite powders were produced by a special fluidized bed process which allowed encapsulation of the solid lubricants with malleable, metallic coatings such as copper, nickel, or silver. Additional work by Hopkins indicated that other coatings, such as CaF2:BaF2 combined with MoS2 and borosilicate glass combined with MoS<sub>2</sub>, appear promising as plasma applied deposits.

Other approaches for the development of better adhesion methods for solid lubricants concerned the use of electrophoresis, electrocodeposition and electroplating techniques.

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In the electrophoretic coating process, a DC potential is applied across two electrodes immersed in a dispersion of colloidal particles suspended in a nonaqueous medium, and the particles are further dispersed to one of the electrodes at which the charge of the electrode is neutralized, and the particles are deposited. Hopkins 8 reported that work on electrophoretic deposition resulted in only moderate success. The following problems were (a) Deposition parameters could be estabencountered: lished only by trial and error. (b) Lubricant or binder may preferentially deposit. (c) Reproducibility of wear Wieser indicated that electrolife results was poor. phoretic deposition as an overcoating process provided additional wear life to (MoSe<sub>2</sub>·TaS<sub>2</sub>·graphite·AlPO<sub>2</sub>) solid films.

Both electrophoretically and electroplated films of  $MoS_2$  were studied as solid lubricant film for instrument bearings by Kirkpatrick. <sup>9</sup> The best performance was obtained with  $MoS_2$  deposited by electroplating.

Electrodepositing of a soft ductile metallic bonding element containing a refractory metal lubricating

element was examined by Thornton. 20 The work indicates that the Au-Mo film system provides the best traits of friction and wear.

Vest $^2$  also used electrodeposition to form a Ni-MoS $_2$  coating on aluminum alloys. Load-carrying capacity on the soft substrate was low and the (dhesion of the film was not reproducible.

The deposition of highly adherent surface films by vacuum methods has unusual promise. Vacuum deposition methods can be divided into two categories: (a) low energy deposition methods (chemical vapor deposition and flash evaporation) and (b) high energy or plasma deposition methods (d-c, r-f sputtering and ion plating). The efficiency and effectiveness of using various high and low energy deposition methods for the application of solid film lubricants was discussed by Spalvins. 24 His observations indicated that the kinetic energy of the impinging metal atoms has a direct effect on the film formation by which, in turn, the strength and the durability of the film are determined Results show that d-c and r-f sputtering techniques can be used to apply solid film lubricants. endurance life of an MoS d-c sputtered film 2,000A thick was greater than five times that of a conventional resin-bonded solid film 130,000Å thick in vacuum environment.

Metallic films (Ni, Au, Ti, Pb) have been deposited successfully by ion plating Spalvins; indicated that strong bonding of the film is established because of the high energy of the plasma (ionized argon) and the high kinetic energy of the depositing material. Friction experiments indicated that, as the ion-plated film wore off, a gradual increase occurred in the coefficient of friction instead of the sharp rise usually observed. Tensile tests show that the ion-plated film plastically flows with the bulk material without peeling or scaling

#### Part III. Composite Lubricants

Since resin-bonded solid lubricants have a finite wear life and must be replaced or repaired, various means have been investigated to lengthen their wear life. Such investigations have resulted in the self-lubricating or composite-lubricant materials. In these materials, solid lubricant powders are incorporated into a basic matrix by vacuum inpregnation, sintering, and high- or low-temperature, high pressure compaction. The basic matrix can be metallic- or

polymeric-fortified with fillers and reinforcement materials for strength. Lubrication is accomplished by a transfer of solid lubricant from within the structure of the composite to the interface between the moving parts. This transfer results in the formation of an effective lubricating film.

A compiled list of recently developed composite lubricants, methods of preparation, and compressive strengths is contained in Table III. Wear rate data on some of the composites listed in Table III are shown plotted in Figure 1. The enclosed areas represent cluster analysis of data instead of individual data points.

Extensive work on metal-carbon fiber composites has been accomplished by Giltrow and Lancaster. The carbon composites containing metals (Pb, Ag, Cu, Ni, Co) have relatively low wear rates but high coefficient of friction values. The addition of a solid lubricant (MoS<sub>2</sub>, NbSe<sub>2</sub>, WSe<sub>2</sub>) does result in lower coefficient of friction values but slightly higher rates of wear.

Composites based on a metal matrix have been investigated by Boes, 15 and the development of self-lubricating composites has been emphasized for use in bearings and gears. These composites were made of two types: (a) a solid lubricant dispersed in a metal matrix (Ag, Cu), and (b) PTFE and a solid lubricant dispersed in a metal matrix (Ag, Cu). The friction and wear characteristics indicated in Figure 1 show that the addition of PTFE reduced the wear rates of the original composites. These composites were successfully used as cage materials in ball bearings at cryogenic temperatures, 400°F and high vacuum (1X10-7Torr).

Hubbell<sup>26</sup> described the fabrication and testing of lubricating composites for use as ball bearing separators. The hot-pressed solid lubricant compacts were composed of a solid lubricant dispersed in a tantalum and cobalt-silver metal matrix. Actual bearing tests conducted showed bearing life greater than 6,700 hours in an air environment at 3,450 rpm with a 10-pound axial load.

Additional work by Campbell<sup>27</sup> was conducted on the development of a lubricating composite for high-speed, high-temperature bearings. Composites were compounded with  $MoS_2$  in a matrix of iron and platinum. These composites were then machined as retainers for both ball and roller bearings. They operated successfully in bearing tests at 15,000 rpm, 680°F in vacuum (1X10<sup>-5</sup> Torr).

TABLE 111

PROPERTIES
AND
PREPARATION.
MATERIALS.
COMPOSITE

Composite	Preparation	Compressive Strength	Legend for Figure 1
The carbon fiber (1)  Ca + carbon fiber (1)  Co + carbon fiber (1)	Hot pressed at 600-1000°C at 1 con/in <sup>2</sup> or compacted at 250°C followed by vacuum sintering at 700°C	8 ton/in? .	~ W M 44 W
Mi + Carbon fiber (1) + MoSz Mi + Carbon fiber (1) + MbSez Mi + Carbon fiber (1) + Mbsez	Hot pressed at 850-900°C	ļ	9 <b>~</b> 8
Aq.C + {MeSez, MoSz, MSez, h.Sez}	Hot pressed	:	
Ag.Cu + (MeSez. MoSz. WSez. NeSez) + PTFF.	Not pressed	ļ	::::::::::::::::::::::::::::::::::::::
Ta + NoSz Ag + Co + MSz	Mot pressed at 1500-3000% at 4000 to 7000 psi	14.900 pst	
Galla + MSea	Compartion at room temperature at 50,000 psi	19,980 ps1 @ 2,075 ps1	71
fe + Pt + MoSz	Hot pressed 1600-2500°F at 7200 psi	į	i
MS + Bafs:Cafs MS:Cr + Bafs:Cafs	Compaction at room temperature at 20,000 ps; followed by sintering at 2150°F	50,000 ps1 80,000 ps1	٤
±3 + 1405 <sub>2</sub>	Hot pressed	25,000 psi @ 11,000 psf	*1
PFE + carbon fiber (1) PFE + carbon fiber (11)	Compression molded at 380°C	: :	212 201
Thermoset + carbon fiber (1) Thermoset + carbon fiber (11)	Compression molded at 300°C		
Polyimide + graphite	Compression molded at 350°F	19.000 pst 0.000 pst	96
Polytatde + MoS <sub>2</sub>	Compression molded	25,000 psf	20
PTFE + Pb + bronze + glass flbers	fitament winding, followed by vacuum impregnation and sintering at 700°F		:

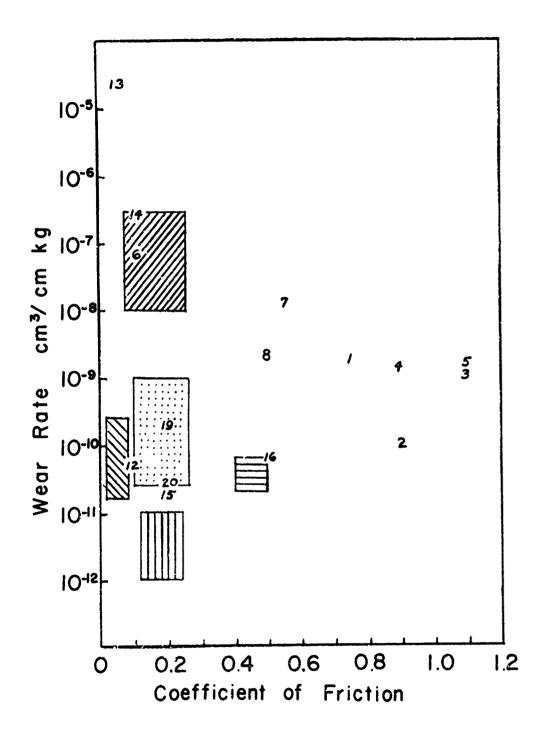


FIGURE 1
COMPARATIVE PERFORMANCE OF VARIOUS
TYPES OF SELF-LUBRICATING COMPOSITES

Weapons Laboratory at Rock Island, Research Directorate.

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A unique solid lubricating composite was developed by  $Boes^{28}$  which resists oxidation at temperatures up to  $1500^{\circ}F$ . These composites were successfully used as retainers for ball bearings capable of long-term operation under high-speed, high-load, and high-temperature conditions. These composites were formulated from  $WSe_2$  - gallium/indium matrix. Major advances in operating life over the former state of the art were achieved by a combination of improvements in retainer design, bearing design, and composite lubricating characteristics.

Other high-temperature lubricating composites were developed by Sliney. These composites consisted of porous nickel and nickel/chromium vacuum impregnated with molten fluorides. In air, the maximum useful service temperature of the nickel composites is about 1100°F; the corresponding temperature for the nickel/chromium composites is about 1350°F. Higher friction coefficients were obtained with these porous metals than with coatings of the same fluoride composition on dense metals; however, low wear and excellent wear life were obtained. The advantages of coatings (lower friction) and of composites (longer wear life) were combined by application of a thin sintered film of eutectic fluoride to the surfaces of the composites

Nickel/MoS<sub>2</sub> composites were developed and examined by Kauzlarich.<sup>30</sup> These composites show high wear rates, but low coefficient of friction values. The areas of application of these composites have not yet been fully developed, nor have their physical and lubricating characteristics been fully established

A considerable amount of work has been done on the use of plastics as self-lubricating materials. Some of the most successful and extensive uses have been in the form of inserts in plain bearings, as reinforced thin sheets on plain spherical bearings and as the retainer material for ball bearings. The most notable plastics used are the nylons, acetals, polytetrafluoroethylene, phenolics, and polypropylene. Fillers and lubricants incorporated to give these materials higher strength and lower friction are glass and ceramic fibers, copper, lead, molybdenum disulfide, and graphite. Discussions on lubricating plastics are given by Campbell<sup>3</sup> and Zimmerman<sup>3</sup> along with information on mechanical properties, comparative performance data, and other pertinent information. Composites of polytetrafluoroethylene (PTFE) containing two types of carbon fiber were prepared and examined for their wear properties by Giltrow and Lancaster.

Type I carbon fiber (partially graphite) composites indicated a lower coefficient of friction and wear rate when compared with Type II (nongraphitic) carbon fiber composites. However, at higher stresses the reverse situation takes place.

In more recent works by Giltrow and Lancaster, 34 the friction wear properties of thermosetting resins reinforced with Type I and Type II carbon fibers were studied. In general, the Type I carbon fiber composites show a lower coefficient of friction and wear rate values than Type II carbon fiber composites. The wear rates of Type II carbon fiber composites are dependent upon the counterface material whereas Type I carbon fiber composites are not. The wear rates of Type I carbon fiber are dependent upon the surface roughness, whereas Type II carbon fiber composites are not.

In more recent work by Devine, <sup>35</sup> the lubricating properties of a new thermuplastic based on aromatic polyimides were investigated. The polyimides are heat stable to 700°F and have good mechanical and electrical insulating properties. The graphite-filled polyimide composites that were developed had a lower wear rate and a lower friction coefficient than the unfilled polymer. However, the mechanical strength is lower than the unfilled polymer. The use of this composite in ball bearings results in a significant increase in parformance life.

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Several commercially available polyimide composites containing PTFE and  $MoS_2$  were studied by Jones. The friction and wear behavior of these composites was studied under sliding conditions, with the use of light loads in both air and vacuum. Polyimide-filled PTFE exhibited lower friction coefficients than  $MoS_2$ -filled polyimides, whereas the lowest rear occurred with composites containing  $MoS_2$ .

Halliwell<sup>3?</sup> has conducted tests with PTFE reinforced with metallic filament windings. The purpose was to develop a piston seal for high pressure air compressors. The filament winding technique resulted in a superior reinforcing matrix when compared with randomly dispersed particles and fibers previously report. Possible reliable compressor operation could be expected at 5000 psi with such seals in lieu of conventional split rings for periods beyond 1000 hours.

.Vest<sup>38</sup> reports on tests in which PTFE-MoS<sub>2</sub> retainers are used in B542 size bearings for spacecraft components. Wer life in excess of 12,000 hours was obtained at high vacuum ( $10^{-7}$  to  $10^{-9}$  Torr).

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### SUMMARY

New developments in the areas of solid lubricant powders, bonded films, and self-lubricating composites have been reviewed along with some new applications.

Generally, the research on new lubricating powders has involved the mechanism of lubrication and the incorporation of these new lubricants into a bonded film or composite. Solid lubricant powders in gas streams have promise, particularly for extreme high-temperature operation or for use in other environmental extremes in which fluid or bonded films would deteriorate.

The bonded solid film lubricants remain the most widely used dry lubrication systems in military and civilian applications. The emphasis on improved wear life and better high-temperature performance continues to be the main objective of research. The synergistic effect of various oxides such as  $Sb_2O_3$  for improving wear life has been supported by many investigators. The development of organic resins such as polyimide, polybenzimidazole, and polybenzothiazole for bonding films has resulted in better high-temperature performance. The success of these films points out the need for keeping abreast of new high-temperature organic materials developed to guarantee the rapid use of these materials in bonded films.

The development of inorganic bonded films has not been so rapid as that of organic films. The use of silicates, phosphates, and fluorides has extended the use of solid film lubricants to temperatures above 1200°F. The use of these lubricants is limited, however, to the range of 500°F to 1200°F because they do not perform so well as the resinbonded films at lower temperatures. In addition, the high curing or fusing temperatures of these lubricants is detrimental to many alloys. Therefore, inorganic bonded films need better low temperature performance, longer wear life, and improved application techniques.

Work on new processes of application has been emphasized because the method of application greatly influences the performance of bonded films. The plasma-spraying process has the most potential for widespread use. This process offers a means of (a) applying solid film lubricants on a production-line basis, (b) applying widely verying types of fibers, and (c) applying high-temperature (ceramic-bonded) solid-film lubricants without seriously affecting the physical properties of alloys.

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The electrophoretic deposition process has resulted in only moderate success; more work needs to be done on establishing the deposition parameters. The process should be considered as a method of enriching or supplementing films applied by other methods.

Vacuum deposition methods of application by use of sputtering and ion-plating techniques offer unique methods with unusual promise. The combined conditions of an extremely clean substrate, high-velocity application of coating material, and ionization of that material provides qualities needed for highly adherent films. Vacuum-deposited films appear to have better wear life in vacuum environments. However, these films are limited to light loads. Therefore, much needs to be resolved before these techniques can be seriously considered for production use.

The composite lubricants offer the most promise of current solid lubricant technology since many more applications are possible with composites. For example, the composites can be machined or formed to various configurations. In addition, with the use of composite lubricants, the problem of finite wear life of bonded films can be solved by provision of a larger reservoir supply of lubricant.

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The metal-based composites have the advantage of higher strength and higher temperature capability when compared with the plastic composites. However, the metal composites still do not have the overall strength when compared with that of bearing materials. So the research emphasis has been on the improvement of mechanical properties through the study of such parameters as pressing pressures, pressing temperatures, and die materials. In addition, more information is needed on design properties such as tensile and compressive strength for selection purposes.

The recently developed plastic composites in which polyimides are used have high strength and are heat stable up to 700°F. The use of graphite and molybdenum disulfide, as solid lubricant additives has improved the wear life of the plastic composites, but has reduced the mechanical strength of these composites. The strength of PTFE-based composites has been considerably improved by use of filament-winding techniques with glass and carbon fibers.

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### CONCLUSIONS AND RECOMMENDATIONS

The successful use of solid lubricants is dependent upon the technique of application and the design of application. Therefore, a need exists for more component test data on properties of the new materials being developed.

This work will lead to a group of recommended lubricating composite systems. As a continuing study, the most applicable areas for composite lubricant use on small arms should be determined.

- Boes, D. J., "New Solid Lubricants: Preparation, Properties and Potentials," Paper presented at 20th Annual ASLE Meeting, Detroit, Michigan, May 4-7, 1965, Preprint 65AM5C3.
- Magie, P. M., "A Review of the Properties and Potentials of the New Heavy Metal Derivative Solid Lubricants," <u>Lubrication Engineering</u>, Vol. 22, No. 7, pp. 262-269, July 1966.
- 3. Devine, M. J., Cerini, J. P., Chappell, W. H., and Soulen, J. R., "New Sulfide Addition Agents for Lubricant Materials," <u>ASLE Transactions</u>, Vol. 11, No. 4, pp. 283-289, October 1968.
- 4. Boes, D. J., and Chamberlain, B., "Chemica! Interactions Involved in the Formation of Oxidation-Resistant Solid Lubricant Composites," ASLE Transactions, Vol. 11, No. 4, pp. 131-139, October 1968.

- 5. Hubbell, R. D., McConnell, B. D., and Van Wyk, J. W., "Development of Solid Lubricant Compacts for Use In Ball Bearing Separators," <u>Lubrication Engineering</u>, Vol. 25, No. 1, pp. 31-39, January 1969.
- 6. Fusaro, R. L., and Sliney, H. E., "Graphite Fluoride (CF<sub>X</sub>)<sub>n</sub> A New Solid Lubricant," <u>ASLE Trans-actions</u>, Vol. 13, No. 1, pp. 56-65, January 1970.
- 7. Gisser, H., Petronio, M., and Shapiro, A., "Graphite Fluoride as a Solid Lubricant," <u>ASLE Proceedings International Conference on Solid Lubrication 1971</u>, American Society of Lubrication Engineers, Pack Ridge, Illinois, 1971, pp. 217-221.
- 8. Campbell, M., and Hopkins, V., "Development of Polyimide Bonded Solid Lubricants," <u>Lubrication Engineering</u>, Vol. 23, No. 7, pp. 288-294, July 1967.

- 9. McConnell, B. D., "Synthesis of Polybenzothiazole Bonded Solid Lubricating Films," Proceedings of the AFML-MR1 Conference on Solid Lubricants, September 9-11, 1969, AFML-TR-70-127, pp. 166-193, July 1970.
- 10. Hopkins, V., Hubbell, R. D., and Lavik, M. T.,
  "Improved High-Temperature Solid Film Lubricants,"
  AFML-TR-71-61, Part I, April 1971.
- 11. Benzing, R. J., and McConnell, R. D., "Wear Behavior of an Air-Drying Methyl Phenyl Silicone Bonded Molybdenum Disulfide Lubricating Film," AFML-TR-70-179, October 1970.
- 12. Hopkins, V. and Gaddis, D., "Friction of Solid Film Lubricants Being Developed for Use in Space Environments," <u>Lubricating Engineering</u>, Vol. 21 No. 2, pp. 52-58, February 1965.

and the second of the control of the

- 13. Wieser, L. E., "Development of a Double-Deposited 1000°F Solid Lubricant," AFML-TR-70-177, October 1970.
- 14. Sliney, H. S., "Lubricating Properties of Some Bonded Fluoride and Oxide Coatings for Temperatures to 1500°F," NASA TND-478, October 1960.
- 15. Lavik, M. T., "The Friction and Wear of Thin, Fused, Fluoride Films," Proceedings of the AFML-MP1 Conference on Solid Lubricants, September 9-11 1959, AFML-TR-70-127, pp. 212-238, July 1970.
- 16. Hopkins, V., Hubbell, R., and Kremith, R., "Plasma Spraying, A New Method of Applying Solid Film Lubricants," <u>Lubrication Engineering</u>, Vol. 24, No. 2, pp. 72-80, February 1968.
- 17. Kremith, R. D., Rosenberry, J. W., and Hopkins, V.,
  "Solid Lubricant Coatings Applied by Plasma
  Spray," American Ceramic Society Bulletin,
  Vol. 47, No. 9, pp. 813-818, September 1968.

- 18. Hopkins, V., Hubbell, R. D., and Lavik, M. T.,
  "Improved High Temperature Solid Film Lubricants,"
  AFML-TR-67-223, Part II, p. 121, February 1969.
- 19. Kirkpatrick, D. L., and Young, W. C., "Solid Lubricants for Instrument Bearings," Proceedings of the AFML-MRI Conference on Solid Lubricants, Sept 9-11, 1969. AFML-TR-70-127, pp. 407-427, July 1970.
- 20. Thornton, H. R., and Wolanski, Z. R., "Lubrication with Electrodeposited Films of Silver-Rhenium and Gold-Molybdenum," <u>Lubrication Engineering</u>, Vol. 23, No. 7, pp. 271-277, July 1967.
- 21. Vest, C. E., and Bazzarre, F. D., "Co-Deposited Nickel-Molybdenum Disulfide," Metal Finishing, Vol. 65, No. 11, November 1967.
- 22. Spalvins, T., "Energetics in Vacuum Deposition Methods for Depositing Solid Film Lubricants," <u>Lubrication Engineering</u>, Vol. 25, No. 11, pp. 436-441, October 1969.
- 23. Spalvins, T., "Bonding of Metal Lubricant Films by Ion Plating," presented at the ASME/ASLE Lubrication Conference, Cincinnati, Onio, Oct 13-15, 1970, Preprint No. 70LC-16.
- 24. Giltrow, J. P., and Lancaster, J. K., "Friction and Wear Properties of Carbon-Fibre-Reinforced Metals," Wear, Vol. 12, No. 2, pp. 91-105, August 1968.
- 25. Boes, D. J., and Bowen, P. H., "Friction-Wear Characteristics of Self-Lubricating Composites

  Developed for Vacuum Service," ASLE Transactions,
  Vol. 6, pp. 192-200 (1963).
- 26. Hubbell, R. D., McConnell, B. D., and Van Wyk, J. W., "Development of Solid Lubricant Compacts for Use in Ball Bearing Separators," <u>Lubrication Engineering</u>, Vol. 25, No. 1, pp. 31-39, January 1969.

- 27. Campbell, M. E., and Van Wyk, J. W., "Development and Evaluation of Lubricant Composite Materials," Lubrication Engineering, Vol. 20, No. 12, pp. 463-469, December 1964.
- 28. Boes, D. J., Cunningham, J. S., and Chasman, M. R.,
  "The Solid Lubrication of Ball Bearings Under
  High Speed High Load Conditions From -225 to
  +1000°F," Lubrication Engineering, Vol. 27,
  No. 5, pp. 150-159; May 1971.

- 29. Sliney, H. E., "Self Lubricating Composites of Porous Nickel and Nickel Chromium Alloy Impregnated with Barium Fluoride Calcium Fluoride Eutectic," presented at 21st ASLE Annual Meeting, Pittsburgh, Pennsylvania, May 2-5, 1966, Freprint 66AM-1CZ.
- 30. Kauzlarich, J. J., "X-Ray Analysis of MoS2 Ni Composites," Proceedings of the AFML-MRI Conference on Solid Lubricants, September 9-11, 1969, AFML-TR-70-127, pp. 337-339, July 1970.

- 31. Campbell, M. E., Loser, J. B., and Sneegas, E.,
  "Solid Lubricants," <u>Technology Survey</u>, National
  Aeronautics and Space Administration, pp. 33-49,
  Report SP-5059, May 1966.
- 32. Timmerman, P. A., "Lubrication in Outer Space A Survey of Literature Including the Aspects of Friction and Wear," Masters Degree Thesis for MS in Department of Mechanical Engineering at University of Iowa, pp. 321-378, June 1964.
- 33. Giltrow, J. P., and Lancaster, J. K., "The Friction and Wear of Carbon Fiber-Reinforced PTFE,"

  Proceedings of the AFML-MR1 Conference on Solid Lubricants, September 9-11, 1969, AFML-TR-70-127, pp. 305-331, July 1970.
- 34. Giltrow, J. P., and Lancaster, J. K., "The Role of the Counterface in the Friction and Wear of Carbon Fibre Reinforced Thermosetting Resins,"

  Wear, Vol. 16, No. 5, pp. 359-374, November 1970.

- 35. Devine, M. J., and Kroll, A. E., "Aromatic Polyimide Compositions for Solid Lubrication," <u>Lubrication Engineering</u>, Vol. 20, No. 6, pp. 225-230, June 1964.
- 36. Jones, J. R., and Gardos, M. N., "friction and Wear Characteristics of Lubricative Composites in Air and in Vacuum," <u>Lubrication Engineering</u>, Vol. 27, No. 2, pp. 47, February 1971.
- 37. Halliwell, H., Ward, J. R., Skruch, H. J., and Thomas, G. L., "An Application of Self Lubricated Composite Materials," <u>Lubrication Engineering</u>, Vol. 23, No. 7, pp. 278-287, July 1967.
- 38. Vest, C. E., and Bowden, W. W., "Evaluation of Space Lubricants Under Oscillatory and Slow Speed Rotary Motion," <u>Lubrication Engineering</u>, Vol. 24, No. 4, pp. 163-167, April 1968.